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**Cc:** CN=Holly Ferguson/OU=CI/O=USEPA/C=US@EPA;CN=Lauren Drees/OU=CI/O=USEPA/C=US@EPA[]; N=Lauren Drees/OU=CI/O=USEPA/C=US@EPA[]  
**From:** CN=Steve Vandegrift/OU=ADA/O=USEPA/C=US  
**Sent:** Wed 7/25/2012 8:05:55 PM  
**Subject:** Fw: Pavillion Phase V ADQ-NERL Las Vegas Lab results follow-up

Rick-

Based on Patrick's response, and the data we have for the matrix spikes, it is my recommendation that this high recovery for the LFB not be used in the evaluation of this data. Six matrix spikes were done, with four having low recovery. The other LFB that was done had low recovery, so the K2 flagging still stands.

I need to make a change to the Recommended Corrective Action for Obs #14e to incorporate this if this action is acceptable to you.

sv

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----- Forwarded by Steve Vandegrift/ADA/USEPA/US on 07/25/2012 02:58 PM -----

**From:** Patrick DeArmond/LV/USEPA/US  
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**Cc:** Rick Wilkin/ADA/USEPA/US@EPA, Holly Ferguson/CI/USEPA/US@EPA, Lauren Drees/CI/USEPA/US@EPA  
**Date:** 07/25/2012 01:09 PM  
**Subject:** Re: Fw: Pavillion Phase V ADQ-NERL Las Vegas Lab results follow-up

Steve,

I really don't know what to make of that abnormally high recovery. Alkylphenols, like the ethoxylates, are ubiquitous in all kinds of products. I know that the SPE cartridges have been known to have trace amounts of them present, but the cartridges are washed extensively prior to extraction to remove any that have been left over from the SPE polymerization reaction during production. And I think I read that number earlier and mistakenly read it as 74.2% instead of 742%, which is why there was nothing mentioned in the report narrative.

There was no spiking error, if so, the octylphenol would also have been exceptionally high, since those two were in the same spiking solution.

That was pretty much the highest we have seen it in any of our samples (apart from 1 or 2 of the Pavilion samples), so I don't think it is in our water source or from our solvents. My hypothesis would be that it came from an SPE cartridge whose SPE resin was improperly polymerized? They all came from the same lot, but I don't know how they are produced.

We might need to look into ordering lot-certified alkylphenol-free cartridges to prevent this happening in

the future.

From my experience with these alkylphenols, they don't always appear to be "well-behaved". There are a lot of factors that contribute to this: their ubiquitousness in many products, they are byproducts of products, notably the ethoxylates, their poor extraction recoveries, and their multiple isomers. I recently talked to Larry Zintek in Reg. 5, who also does work with alkylphenols, and he concurred with that statement about them not being well-behaved.

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Cc: Patrick DeArmond/LV/USEPA/US@EPA, Holly Ferguson/CI/USEPA/US@EPA, Lauren Drees/CI/USEPA/US@EPA  
Date: 07/24/2012 07:33 AM  
Subject: Re: Fw: Pavillion Phase V ADQ-NERL Las Vegas Lab results follow-up

Rick/Patrick-

For nonylphenol, the LFB on 4/20 had a recovery of 742%. The same sample had 127% recovery for octylphenol. There is nothing in the report narrative about this high recovery. Was it determined why this one had such high recovery? It is certainly not consistent with the rest of the data. Was there a spiking error?

Steve

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Date: 07/24/2012 08:06 AM  
Subject: Fw: Pavillion Phase V ADQ-NERL Las Vegas Lab results follow-up

Hi Patrick - thanks for the quick response. Will let you know if any other follow up is needed.

Rick

----- Forwarded by Rick Wilkin/ADA/USEPA/US on 07/24/2012 08:04 AM -----

From: Patrick DeArmond/LV/USEPA/US  
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Date: 07/23/2012 06:08 PM  
Subject: Re: Fw: Pavillion Phase V ADQ-NERL Las Vegas Lab results follow-up

Hi Rick,

Sorry, these questions were probably a result of me not providing the adequate information. Thanks for your patience, I know you guys are under a lot of pressure.

See answers below in red.

If you need anything else, please let me know, I'll provide it as soon as possible.

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Cc: Steve Vandegrift/ADA/USEPA/US@EPA  
Date: 07/23/2012 12:51 PM  
Subject: Fw: Pavillion Phase V ADQ-NERL Las Vegas Lab results follow-up

Hello Patrick - a couple of questions came up during the ADQ process. Can you have a look at the questions below and get back to us? Thanks much.

Rick

=====  
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Date: 07/23/2012 02:38 PM

Subject: Pavillion Phase V ADQ-NERL Las Vegas Lab results follow-up

Rick-

There are several items needing either clarification or additional information as a result of the recent ADQ on the Phase V data. These questions should be forwarded to the Las Vegas laboratory.

1. Holding Time: It was noted that several samples were re-extracted. These re-extractions were beyond the 30 day holding time. Were any data from these re-extractions used in the final data report? If so, which ones? Yes, certain samples were re-extracted for alkylphenol or AEO analysis. Halfway through MS analysis, the mass spec that we were previously using to analyze for alkylphenols needed repair and wasn't functional. Hence, we transferred method over to new MS, but also needed to re-extract some samples due to lack of sample. Re-extractions were performed on 5/21/12 and 6/1/12. On 5/21/12, the samples that were past 30 days were LV12WAT058 (equipblk1), LV12WAT060 (PGDW20-0412), and LV12WAT062 (PGDW20d-0412). Alkylphenol data from these samples were used in the final results. Then in the 6/1/12 re-extraction, performed because some analytes were very high concentrations, LV12WAT064 (EPAMW02-0412-1) alkylphenol data was used in the final results (prior extraction provided results that exceeded calibration range).

To summarize, equipblk1, PGDW20-0412, PGDW20d-0412, and EPAMW02-0412-1 alkylphenol data were used in final data report, and these had been re-extracted past 30 day hold time.

2. Acrylamide: It does not appear that a laboratory-fortified blank was analyzed with the batch on 5/1/2012, the QAPP specifies one is to be analyzed with each batch. I looked at the batch from 5/1/12, and it appears that that is correct, no LFB was analyzed with the 5/1/12 batch. I interpreted the "batch of samples" to refer to all the samples we received from your sampling trip. Because I can only extract 6 samples at a time with our Autotrace SPE Workstation, I often analyzed the extracted samples on the MS on different days. The batch did include a full initial calibration and continuing calibration verification and a number of sample matrix spikes. The concentration of the spike solution is not clear from the run logs (these were spiked at 500 ng per 500 mL, so final concentration of 1 ppb, I can provide missing pages from run logs if needed); therefore matrix spike recovery values are not known for those samples. The laboratory should provide spike recovery values for all spikes (LFB and matrix spike) for each analytical batch (5/1, 5/14, and 5/15) and identify which samples correspond to each spike recovery.

4/26/12 LFB spike recovery = 114%

5/1/12: LV12WAT060 = PGDW20-0412, spike recovery = 102%.

5/1/12: LV12WAT062 = PGDW20d-0412, spike recovery = 129%.

5/1/12: LV12WAT066 = PGDW23-0412, spike recovery = 126%.

5/1/12: LV12WAT073 = PGDW05-0412, spike recovery = 115%.

5/14/12 LFB spike recovery = 106%

5/14/12: LV12WAT088 = EPAMW02-0412 spike recovery = 115%

5/15/12: LFB spike recovery = 120%

5/15/12 LV12WAT105 = Riverton WY truck water spike recovery = 118%

3. Alkylphenols. (a) Spike recoveries are listed in a column but it is not apparent which samples they are associated with (LFB or matrix spike). The laboratory should identify which samples correspond to each spike recovery.

For nonylphenol:

LFB 5/21/12: 63.5%

LV12WAT060 (PGDW20-0412) spike 5/21/12: 69.2%

LV12WAT062 (PGDW20d-0412) spike 5/21/12: 62.8%

LV12WAT060 (PGDW20-0412) spike 4/25/12: 57.6%

LV12WAT062 (PGDW20d-0412) spike 4/25/12: 70.9%

LV12WAT073 (PGDW05-0412) spike 4/25/12: 56%

LV12WAT105 (Riverton water) spike 5/11/12: 107%

LFB 4/20/12: 742%

For octylphenol:

LFB 5/21/12: 109%

LV12WAT060 (PGDW20-0412) spike 5/21/12: 116%

LV12WAT062 (PGDW20d-0412) spike 5/21/12: 120%

LV12WAT060 (PGDW20-0412) spike 4/25/12: 110%

LV12WAT062 (PGDW20d-0412) spike 4/25/12: 117%  
LV12WAT073 (PGDW05-0412) spike 4/25/12: 105%  
LV12WAT105 (Riverton water) spike 5/11/12: 126%  
LFB 4/20/12: 127%

(b) It appears that the values for samples EPAMW02-0412-1 (both analytes) and octylphenol for sample EPAMW02-0412-2 are above the calibration range. Were these samples diluted and re-run to be within the calibration range?

The samples were quantitated using isotope dilution technique for alkylphenols, based on relative response between native and labeled compound. Diluting the samples would not change the ratio between native and isotope, therefore, EPAMW02-0412-1 was re-extracted on June 1, 2012 (only 50 mL was extracted instead of 500 mL). My student extracted all the samples and accidentally forgot to add labeled compound to the last EPAMW02-0412-2 sample, therefore, it could not be re-extracted one more time.

Steve

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